CORE
Metadata, citation and similar papers at core.ac.uk
Provided by University of Fukui Repository

Journal of Template
Vol. 00, No. 0, 0000

Effect of Forming Conditions on Crystallization in Laser Forming of Palladium Based Thin Film Metallic Glass*

Masaaki OTSU** Yuki IDE*** Mitsuhiro MATSUDA***
and Kazuki TAKASHIMA***
**Department of Mechanical Engineering, University of Fukui,
3-9-1, Bunkyo, Fukui, 910-8507, Japan
E-mail: otsu@u-fukui.ac.jp
***Department of Materials Science, Kumamoto University,
2-39-1, Kurokami, Kumamoto, 860-8555, Japan

Abstract
$Pd_{77}Cu_6Si_{17}$ thin film metallic glasses of which thicknesses were 10, 20 and 28 μm were bent by laser forming. The working conditions were changed and crystallization of thin films was investigated. When the laser power was changed under fixing the scanning velocity to 40 mm/s and the Q-switch frequency to 3 kHz, crystallization was observed at laser irradiated surface and/or the reverse side to the laser irradiation. In a case of changing the scanning velocity under fixing the Q-switch frequency to 3 kHz and the laser power to 2.0 W for the film thickness of 20 μm and 3.0 W for the film thickness of 28 μm, the working conditions without crystallization were found at high scanning velocity. When the Q-switch frequency was changed under fixing the scanning velocity to 40 mm/s and the laser power to 2.0 W for the film thickness of 20 μm and 3.0 W for the film thickness of 28 μm, the working conditions without crystallization were found at low Q-switch frequency.

Key words: Laser Forming, Micro Bending, Forming Conditions, Metallic Glass, Thin film, Crystallization, MEMS

1. Introduction

Electric devices and semiconductor devices having lighter weight and higher performance are required for appliances and automotive sensors and actuators. MEMS (Micro Electro Mechanical Systems) devices are promising. For enhancing the reduction of the device size and weight, and improvement of the performance, it is necessary to fabricate them with not only two-dimensional but also three-dimensional structure in micrometer order. Since conventional fabrication methods for microdevices are, however, layer integration process like lithography, it is difficult to manufacture three-dimensional structure with large height. Therefore new forming methods are required for making microdevices having three-dimensional structure with large height, and plastic working methods, especially bending of thin films, are very efficient ones.

From the view point of microdevice materials, crystalline materials like single crystalline or polycrystalline silicon are mainly used for microdevices. For further miniaturization of microdevices, the anisotropy of those materials becomes serious issue in fabrication and performance. Therefore amorphous materials are under discussion for the next generation MEMS devices. Metallic glasses have less size effect and anisotropy, and it

*Received XX Xxx, 200X (No. XX-XXXX)
[DOI: 00.0000/ABCDE.2006.000000]

is one of the promising materials for the next generation MEMS devices. It is, however, difficult to fabricate the microdevices having large aspect ratio three-dimensional shape by the conventional wafer processes. The authors suggested using laser forming to bend thin film metallic glass to make large aspect ratio device [1]. Although no crystallization was observed in the paper, the number of investigated working parameters was not so large. More detailed investigation for clarifying the relations between the working conditions and crystallization after laser forming is necessary.

In this study, $Pd_{77}Cu_6Si_{17}$ thin film metallic glasses were bent by laser forming and relation between the working conditions and crystallization after laser forming was studied.

Nomenclature

N : scanning number
P : laser power, W
F : Q-switch frequency, kHz
V : laser scanning velocity, mm/s
α : diffraction angle, °
θ : bending angle, °

2. Experimental method

2.1. Specimen

$Pd_{77}Cu_6Si_{17}$ thin film metallic glass [2] used for specimen was fabricated by sputtering on a silicon wafer and removed from the silicon wafer by peeling mechanically. The thin films were cut into 10 mm in length and 1.45 mm in width by a knife. The dispersion of thickness was from -2 to +2 µm. Because of residual stress introduced by sputtering, the thin films were a little distorted. Laser was irradiated on the sputtered surface. The specimens were analyzed by XRD and DSC before laser irradiation to check the amorphous state. From the analyses, the glass transition temperature was confirmed to 623 K and the crystallization temperature was 680 K.

2.2. Setup

Experimental setup is illustrated in Fig. 1. A 50 W Nd-YAG laser was employed. The specimen was fixed to a holder on an NC table. Switching on/off and setting the laser power, scanning velocity and Q-switch frequency were controlled by a personal computer. Before and after laser irradiation, the specimen was moved to a laser displacement sensor and the bending angle was measured.

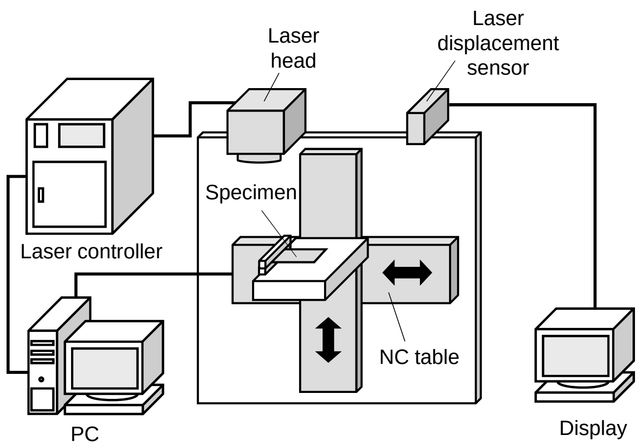


Fig. 1 Experimental apparatus for laser forming of thin film metallic glass.

2.3. Procedure

Laser forming was performed at just focused position and the laser power, scanning velocity, scanning number and Q-switch frequency were changed. The working conditions are shown in Table 1. After laser forming, the bending angle was measured and XRD analysis was carried out. In XRD analysis, the laser irradiated and reverse surfaces were measured. X ray was irradiated at the center (0.72 mm x 0.18 mm) and border (0.03 mm x 0.18 mm) positions of the laser irradiation. A diameter of X ray was 100 μm and enough smaller than that of laser beam of 365 μm. This means the XRD analysis can be evaluate only laser irradiated area except the substrate and the heat affected zone.

Table 1 Laser forming conditions.

Operation mode	Q-switch Pulsed / CW
Wavelength [nm]	1064
Q-switch frequency, *F* [kHz]	1-60, CW
Laser power, *P* [W]	1-7
Scanning velocity, *V* [mm/s]	40-65
Scanning number, *N*	1-80
Defocus length, [mm]	0
Atmosphere	Air

3. Results

3.1. Effect of laser power

Relation between the bending angle, θ, and the laser power, *P*, after laser forming at $V = 40$ mm/s in scanning velocity, $F = 3$ kHz in Q-switch frequency and $N = 50$ in scanning time is shown in Fig. 2. The thicknesses of thin film were $t = 10$, 20 and 28 μm. The required laser power for obtaining enough bending angle decreased as the thickness of thin film decreased.

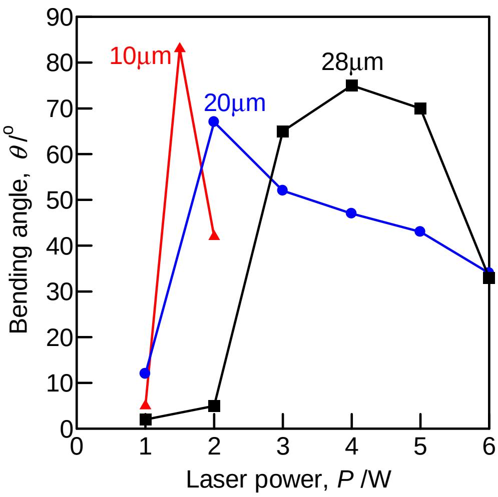
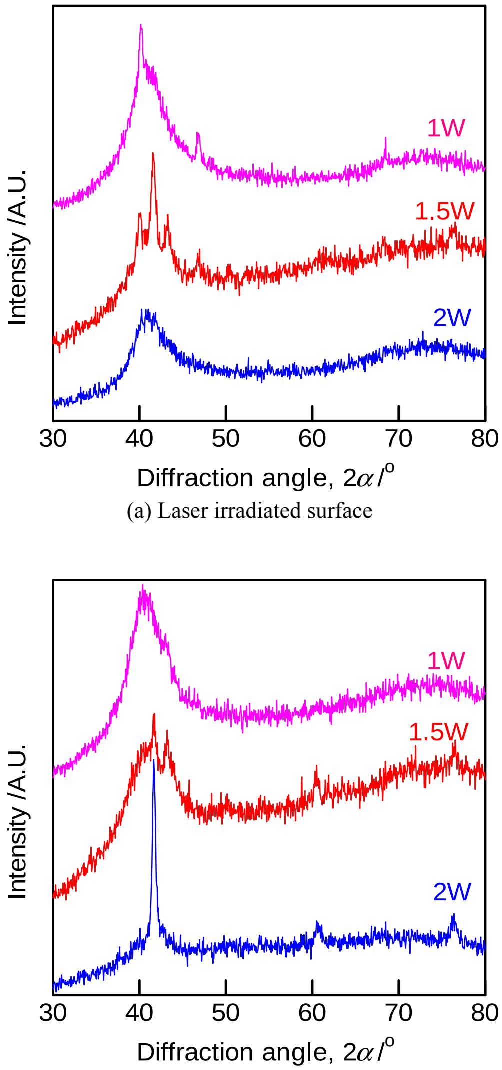


Fig. 2 Relations between bending angle and laser power of various thickness for $Pd_{77}Cu_6Si_{17}$.
($V = 40$ mm/s, $F = 3$ kHz, $N = 50$)

Results of XRD analysis of both laser irradiated and reverse surfaces with changing the laser power are shown in Figs. 3 and 4. In cases of the thicknesses of $t = 10$ μm and $t = 20$ μm, the laser irradiated surfaces were crystallized and the reverse surfaces remained amorphous state at $P = 1.0$ W in the laser power. The reason is considerable as follows. The laser power was too small to melt the specimen surface, and both the laser irradiated surface and the reverse surface kept solid state. The nose position in the TTT diagram of metallic glass in cooling at solid state moves to shorter time than that in cooling from liquid state to solid state. Since the laser irradiated surface was heated with keeping solid state, it was crystallized, and since the temperature elevation of the reverse surface was lower than the crystallization temperature, it remained amorphous state.



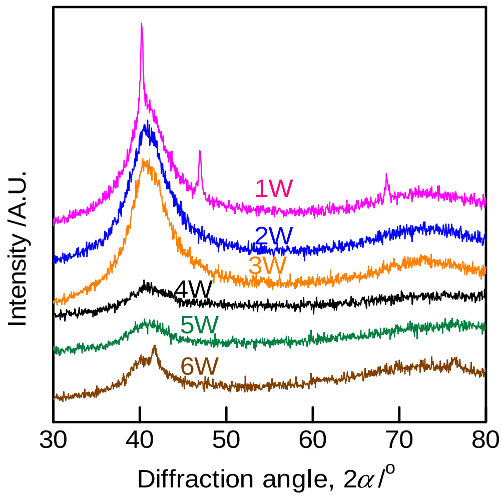
(a) Laser irradiated surface

(b) Reverse surface

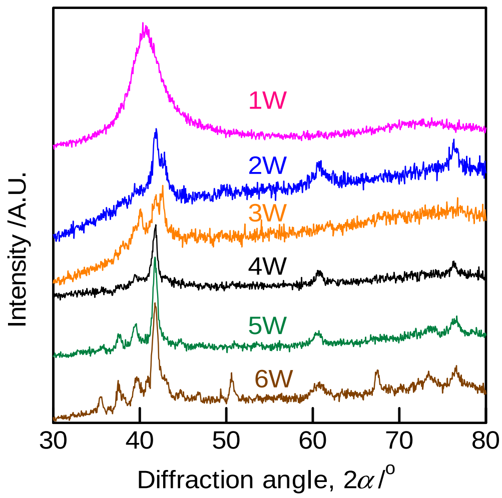
Fig. 3 XRD patterns of laser irradiated zone. (t = 10 µm, V = 40 mm/s, F = 3 kHz, N = 50)

When the film thickness was t =10 µm and the laser power was P = 2.0 W, and when the film thickness was t =20 µm and the laser power was P = 2.0 to 5.0 W, the laser irradiated surface was amorphous state and the reverse surface crystallized. This is because the laser irradiated surface melted and the cooling rate was faster than the critical cooling rate. The cooling rate in the reverse surface was, however, slower than the critical cooling rate since it requires higher value due to solid state cooling. The critical cooling rate of $Pd_{77}Cu_6Si_{17}$ was not found in literatures but that of $Pd_{78}Cu_6Si_{16}$ is from $10^{2.4}$ to $10^{2.8}$ K/s [(3)].

When the scanning velocity, the Q-switch frequency and the scanning number were fixed to V = 40 mm/s, F = 3 kHz and N = 50, respectively, enough bending without crystallization could not be obtained.



(a) Laser irradiated surface



(b) Reverse surface

Fig. 4 XRD patterns of laser irradiated zone. (t = 20 µm, V = 40 mm/s, F = 3 kHz, N = 50)

3.2. Effect of scanning velocity

The laser power, the Q-switch frequency and the scanning number was set to P = 1.5 W (t = 10 µm) , 2.0 W (t = 20 µm), 3.0 W (t = 28 µm), F = 3 kHz and N = 50, respectively, and laser forming was performed by changing the scanning velocity. Relation between the bending angle and scanning velocity is shown in Fig. 5. As the scanning velocity increased, the bending angle decreased. When the film thickness of the specimen became smaller, large bending angles were obtained in spite of large scanning velocity.

Figures 6 and 7 show the results of XRD analysis of laser irradiated specimens. In the case of t = 10 µm in the thickness and V = 40 and 45 mm/s in the scanning velocity, both the laser irradiated and reverse surfaces crystallized. In the case of t = 20 µm in the thickness, only the reverse surface crystallized. When the film thickness was small, temperature difference between the laser irradiated and the reverse surfaces became small, and both surfaces crystallized because the cooling rate was smaller than the critical one. When the scanning velocity was greater than V = 55 mm/s, the cooling rate was considerable to be larger than the critical one in the both surfaces and the both surfaces kept amorphous state.

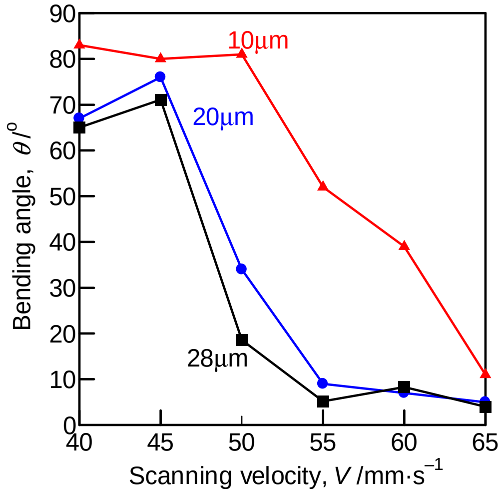
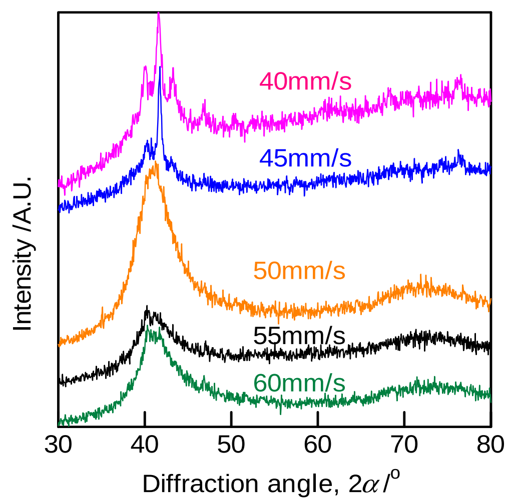
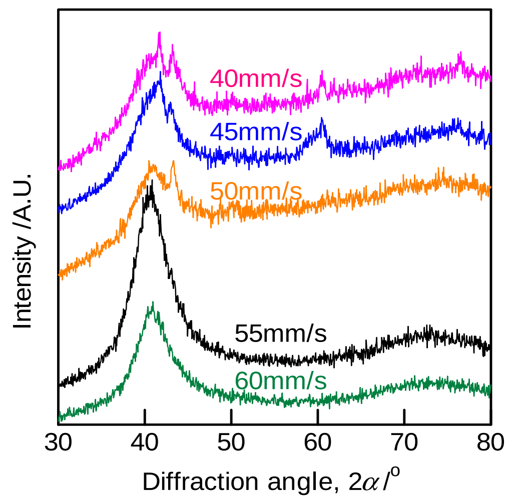


Fig. 5 Relations between bending angle and scanning velocity of $Pd_{77}Cu_6Si_{17}$. (P = 1.5 W (t = 10 µm), 2.0 W (t = 20 µm), 3.0 W (t = 28 µm), F = 3 kHz, N = 50)

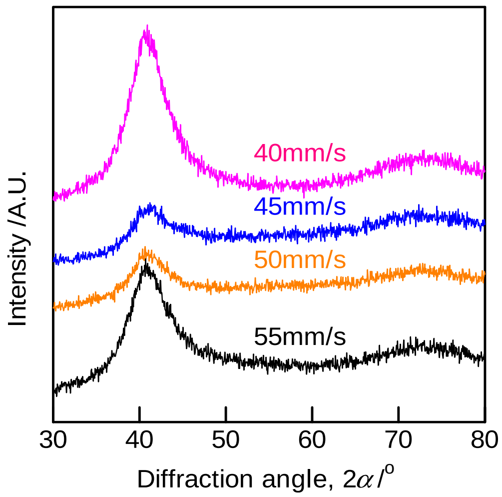


(a) Laser irradiated surface

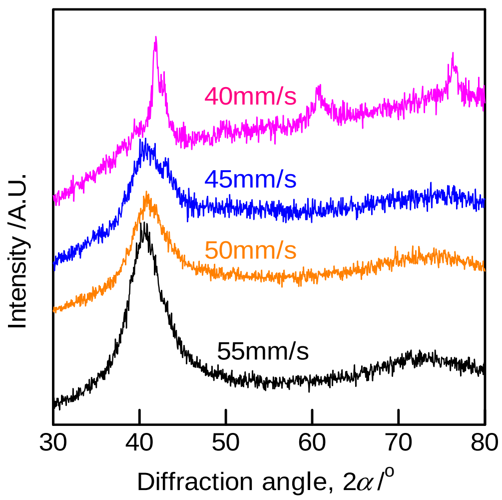


(b) Reverse surface

Fig. 6 XRD patterns of laser irradiated zone. (t = 10 µm, P = 1.5 W, F = 3 kHz, N = 50)



(a) Laser irradiated surface



(b) Reverse surface

Fig. 7 XRD patterns of laser irradiated zone. (t = 20 µm, P = 2.0 W, F = 3 kHz, N = 50)

3.3. Effect of Q-switch frequency

The laser power, the scanning velocity and the scanning number were fixed to P = 2.0 W (t = 20 µm), 3.0 W (t = 28 µm), V = 40 mm/s and N = 50, respectively, and laser forming was carried out with changing the Q-switch frequency. Relation between the bending angle and the Q-switch frequency is shown in Fig. 8. When the Q-switch frequency was less than 2 kHz, the bending angles were very small in both film thicknesses of t = 20 and 28 µm. In the case of 28 µm in thickness, the bending angle was positive at less than 5 kHz in the Q-switch frequency, however, that became negative at 5.5 kHz in the Q-switch frequency. When the film thickness was t = 20 µm, positive bending angles were obtained at less than 60 kHz in the Q-switch frequency and negative ones were gained at continuous wave mode.

Figure 9 shows the result of XRD analysis of 20 µm thickness specimens when the laser power was P = 2.0 W, the scanning velocity was V = 40 mm/s and the scanning number was N = 50, and the Q-switch frequency was changed. When the Q-switch frequency was small, both the laser irradiated surface and the reverse surface were amorphous state. This is because when the Q-switch frequency was small, the peak power became very high and the specimen was heated instantaneously, and distance between each laser irradiated point became long, the cooling rate was larger than the critical one.

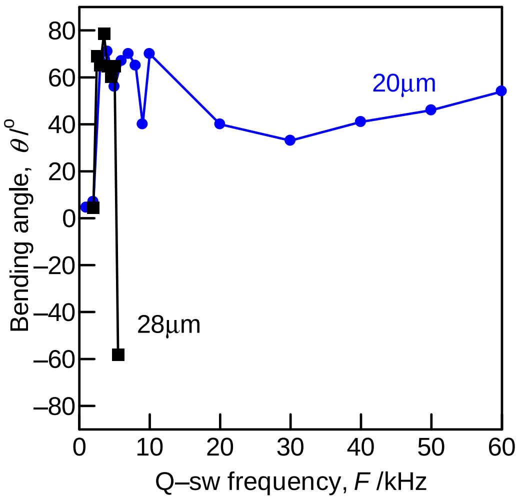
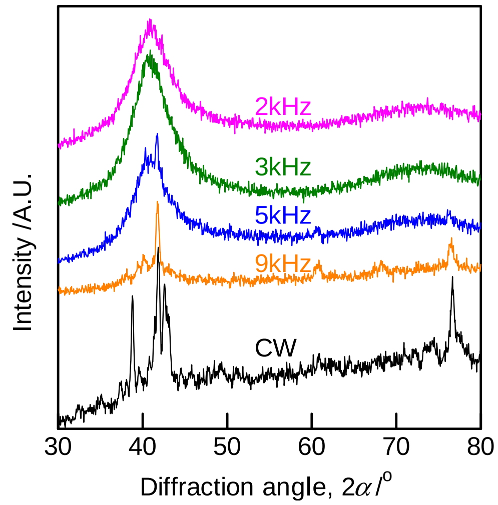
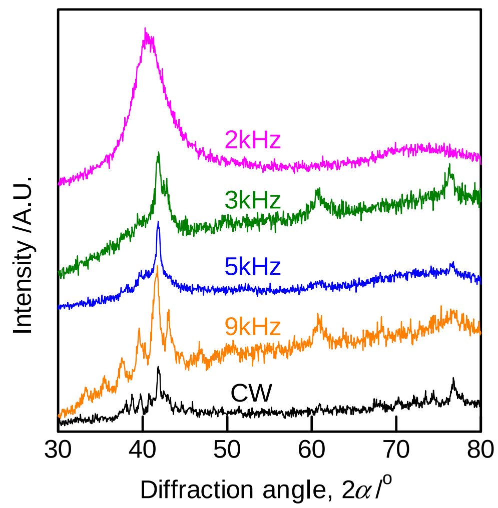


Fig. 8 Relations between bending angle and Q-switch frequency of $Pd_{77}Cu_6Si_{17}$. (P = 2.0 W (t = 20 µm), 3.0 W (t = 28 µm), V = 40 mm/s, N = 50)



(a) Laser irradiated surface



(b) Reverse surface

Fig. 9 XRD patterns of laser irradiated zone. (t = 20 µm, P = 2.0 W, V = 40 mm/s, N = 50)

4. Conclusions

In this study, $Pd_{77}Cu_6Si_{17}$ thin film metallic glasses with the film thicknesses of 10, 20 and 28 μm were bent by laser forming, and the effect of working conditions on crystallization was investigated. Although crystallization at both or one of the laser irradiated surface and the reverse surface was observed with some working conditions, the appropriate working conditions for obtaining enough bending angle without crystallization were confirmed.

Acknowledgments

$Pd_{77}Cu_6Si_{17}$ thin film metallic glass for the specimens were supplied from Prof. Hata at Tokyo Institute of Technology. We express great thank to him. A part of this work was supported by Grant-in-Aid for Young Scientists (B) 19760513.

References

(1) Otsu, M., Ide, Y., Sakurai, J., Hata, S. and Takashima, K., Laser Forming of Thin Film Metallic Glass, *Journal of Solid Mechanics and Materials Engineering*, Vol. 3, No. 2(2009), pp. 387-396.

(2) Yamauchi, Y., Hata, S., Sakurai, J., and Shimokohbe, A., Combinatorial search for low resistivity Pd-Cu-Si thin film metallic glass compositions, *Japanese Journal of Applied Physics*, Vol. 45, No. 2(2006), pp. 5911-5919.

(3) Nishi, Y., Kayama, N., Kiuchi, S., Suzuki, K., and Masumoto, T., Viscosities and Glass Formation of $Pd_{84}Si_{16}$ Alloy and $Pd_{78}Cu_6Si_{16}$ Alloy, *Journal of Japan Institute of Metals*, Vol. 44, No. 12(1980), pp. 1336-1341. (In Japanese)